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2. Patent application number *(The Patent Office will fill in this part)* — 0323229.5 — 3 OCT 2003

3. Full name, address and postcode of the or of each applicant *(underline all surnames)* —
UNIVERSITY OF BRISTOL
Senate House
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BRISTOL
BS8 1TH

   Patents ADP number *(if you know it)* — 79818013.

   If the applicant is a corporate body, give the country/state of its incorporation — UNITED KINGDOM

4. Title of the invention — CANTILEVER PROBE FOR AN ATOMIC FORCE MICROSCOPE

5. Name of your agent *(if you have one)* — STEVENS HEWLETT & PERKINS

   "Address for service" in the United Kingdom to which all correspondence should be sent *(including the postcode)* —
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20/23 Holborn
LONDON
EC1N 2JD

   Patents ADP number *(if you know it)* — 1545003

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Agents for the Applicant

12. Name and daytime telephone number of person to contact in the United Kingdom DR CEILI WILLIAMS; 020 7404 1955

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CANTILEVER PROBE FOR AN ATOMIC FORCE MICROSCOPE

This invention relates to the field of atomic force microscopes, to cantilever probes therefor and to a method of operating such microscopes. In particular, it relates to an atomic force microscope that is operable in constant height mode.

The atomic force microscope (AFM), or scanning force microscope (SFM), was invented in 1986 by Binnig, Quate and Gerber. Like all other scanning probe microscopes, the AFM is based on the principle of mechanically scanning a nanometric probe over a sample surface in order to acquire an "interaction map" of the sample. The interaction force in this case is simply the molecular interaction between the sample and the tip of a sharp probe attached to a cantilever spring. When the probe tip is brought into close proximity with the sample, the cantilever bends in response to the molecular interaction force. Images are collected by scanning the sample relative to the probe and measuring the deflection of the cantilever as a function of lateral position. An optical lever technique is usually used to measure this bending. Since the cantilever obeys Hooke's Law for small displacements, the interaction force between the tip and the sample can be deduced.

The AFM is usually operated in one of two modes. In constant force mode, feedback enables a positioning piezoelectric driver to move the sample (or probe) up or down in response to any change in the interaction force that is detected. In this way, the interaction force may be held relatively steady and a fairly faithful topographical image of the sample is obtained. Alternatively the AFM may be operated in constant height mode. No, or very little, adjustment of the vertical height of the sample or probe is imparted during the scan. In this context, adjustment of the vertical height means that a translation is applied either to an actuator connected to the

cantilevered probe or to the sample itself. There remains therefore a degree of freedom for the probe tip to move up and down as the degree of cantilever bend is varied. In constant height mode, topographical changes to the sample are indistinguishable from interaction force variations in that either or both will cause the cantilever spring to bend.

In addition to these differing feedback regimes, image contrast is usually obtained in one of three different ways. In contact mode the tip and sample remain in close contact, i.e. in the repulsive regime of the molecular interaction, as scanning proceeds. In tapping mode an actuator drives the cantilever in a "tapping" motion at its resonant frequency. The probe tip therefore only contacts the surface for a very small fraction of its oscillation (tapping) period. This dramatically shortened contact time means that lateral forces on the sample are very much reduced and the probe is therefore less destructive to the specimen as the scan is taken. It is consequently much used for imaging sensitive biological specimens. Oscillation amplitude is generally held constant using a feedback mechanism. In non-contact operation the cantilever is oscillated above the sample at such a distance that the molecular interaction force is no longer repulsive. This mode of operation is however very difficult to implement in practice.

Recent advances in probe microscopy have led to much faster data collection times. With faster scan techniques, such as that described in PCT patent application publication number WO 02/063368, finite probe responsivity is increasingly becoming a limiting factor in image collection times. The probe will not respond instantaneously to a change in sample characteristics and so there is an inherent time delay between, for example, the probe encountering a region of the sample surface with increased height and the system reacting to it. This disadvantage applies to both constant force and constant height modes of AFM operation. It is less severe in constant height mode, which is therefore the preferred mode

of operation for fast scanning techniques, but it is still sufficient to limit unduly the scan speed of the current generation of fast scanning probe microscopes.

In constant force AFM mode, an electronic feedback mechanism is usually employed in order to keep the average interaction force constant. As the scan progresses if there is a change in interaction force (for example caused by a change in sample height) this is first observed by the detection electronics, a feedback signal is then generated, the probe or sample height adjusted in response to this signal and then there is also a finite time taken for the probe (or sample) to settle at its new position. This sequence imposes a limitation on the ultimate speed with which a full image scan can be collected.

The problem is not so restrictive if operating in constant height mode, in which electronic feedback is not normally employed to the extent that it is used in constant force AFM. For the interaction force to be measured accurately however the probe tip should, as far as possible, track the contours of the sample surface. This is ensured by exploiting the reaction force developed as the cantilever is bent by the sample surface. That is, as a high region of the sample surface is scanned, the cantilever is increasingly bent upwards and the energy stored in the spring is increased. As the height falls away, a restoring force pushes the cantilever back towards its equilibrium (straight) position, thus maintaining contact with the surface. If however the scan speed is too fast, the probe will not track the surface but will effectively be thrown upwards over any protuberance from the surface and may start to resonate, or "ring". This in turn gives rise to oscillations in the imaged interaction force.

WO 02/063368, referred to above, describes a scanning probe microscope in which the probe is oscillated at resonance whilst translated in order to interrogate a sample very rapidly with an arrangement of scan lines. The

typical time spacing between pixels is therefore shorter than $1/f_r$, where f_r is the resonant frequency of the probe. On the other hand the time taken (τ_{res}) to respond to a change in topography of the sample surface is based on the effective mass of the probe and the spring constant of the cantilever. If $\tau_{res} > 1/f_r$, then clearly the interaction force will not be measured accurately from scan line to scan line, and certainly not for all image pixels.

There is a perceived need to provide for improved probe responsivity to sample topographic fluctuations or to variations in the interaction force and so to permit AFM microscopy to be performed at faster scanning speeds before image artefacts such as those caused by probe ringing start to degrade image quality.

The present invention provides a probe for use in an atomic force microscope or for nanolithography, the probe comprising a cantilever and sharpened tip characterised in that the cantilever is coated on at least one side by an energy-absorbing material.

In an alternative aspect the present invention provides a probe for use in an atomic force microscope or for nanolithography, the probe comprising a cantilever and sharpened tip characterised in that the probe also includes a magnetic element located on the cantilever in the region of the tip.

In a third aspect the present invention provides an atomic force microscope for imaging a sample in accordance with a molecular interaction force between the sample and a cantilever probe, the microscope comprising

driving means arranged to provide relative scanning motion between the probe and the sample surface and capable of bringing the sample and probe into close proximity, sufficient for a detectable interaction to be established between them; and

a probe detection mechanism arranged to measure deflection of the cantilever probe;

characterised in that, the microscope includes the probe as described above.

Alternatively, the microscope is characterised in that it includes direct force (F_{direct}) generating means arranged such that, in operation, a direct force (F_{direct}) is applied to the sample, the probe or between the two, the force (F_{direct}) being directed so as to attract the probe towards the sample or *vice versa*.

In a further aspect the present invention provides a method of collecting image data from a scan area of a sample with nanometric features wherein the method comprises the steps of:-

(a) Moving a cantilever probe coated on at least one side with an energy-absorbing material and with a tip of sub-nanometric dimensions into close proximity with a sample in order to allow an interaction force to be established between probe and sample;

(b) Permitting a direct force (F_{direct}) to be established between sample and probe such that the probe is encouraged to move towards the sample or *vice versa*;

(c) Scanning either the probe across the surface of the sample or the sample beneath the probe whilst providing a relative motion between the probe and surface such that an arrangement of scan lines covers the scan area;

(d) Measuring deflection of the cantilever probe; and

(e) Processing measurements taken at step (d) in order to extract

information relating to the nanometric structure of the sample.

Embodiments of the invention will now be described by way of example only and with reference to the accompanying drawings.

Figure 1 is a diagrammatic illustration of the forces involved as a cantilevered probe makes contact with a sample surface in a prior art atomic force microscope.

Figure 2a shows a schematic implementation of an atomic force microscope that includes a cantilever probe, which is in accordance with a first embodiment of this invention.

Figure 2b shows a schematic implementation of an atomic force microscope that includes a cantilever probe, which is in accordance with a second embodiment of this invention.

Figure 3 is a diagrammatic illustration of the forces involved as a cantilevered probe makes contact with a sample surface in the AFM of Figures 2a and 2b.

With reference to Figure 1, there is shown a sample 1 that is being scanned by a probe of an atomic force microscope (AFM). The probe comprises a substrate 2 from which a cantilever 3 extends, the cantilever 3 having a sharp probing tip 4 mounted at an end remote from the substrate 2. In preparation for a scan, a downwards force ($F_{external}$) is applied to the probe at its substrate end 2 via its mounting to the AFM, moving the probe tip 4 into contact with the sample 1. In order to maintain contact for the duration of a scan, the force $F_{external}$ is greater than that required simply to bring the tip 4 into contact with the sample 1. As a result the cantilever 3 is bent upwards from its rest position 5 as the sample is scanned.

The cantilever 3 obeys Hooke's Law for small displacements. Accordingly

if, when pressing on the sample, the degree of bending is such as to move the tip 4 a perpendicular distance *x* from its rest position and the cantilever spring constant is *k* then the restoring force exerted by the cantilever is *kx*. The downward force exerted by the tip 4, holding it in position tracking the surface, is thus proportional to *kx*.

Clearly the responsivity of the probe tip 4 and hence the resolution of the AFM technique depends on the degree of force *kx* exerted by the cantilever 3 on the sample 1. The greater the force between probe and surface, the greater the responsivity to surface variations. This indicates that a high spring constant *k* is desirable, particularly if the scan is to be fast. On the other hand, the greater the force, the more likely the probe is to damage the sample. Accordingly prior art AFM cantilever probes must make a fundamental compromise between probe responsivity and the likelihood of damaging the sample.

Figure 2a shows a schematic implementation of an AFM, indicated generally by 10, that utilises a first embodiment of a cantilever probe constructed in accordance with an aspect of this invention. The AFM apparatus 10 shown comprises a plate 12 adapted to receive a sample 14, and which is mounted on one prong of a tuning fork 16. The tuning fork 16 is connected to a piezoelectric transducer 18 and a coarse driving means 20. The piezoelectric transducer 18 is used to drive the sample 14 (together with the plate 12 and fork 16) in three dimensions: *x*, *y* and *z* directions. As is conventional in the field, the *z* axis of a Cartesian coordinate system will be taken to be that perpendicular to a plane occupied by the sample 14. That is, the interaction force is dependent both on the *xy* position of a probe 22 over the sample 14 (the pixel it is imaging), and also on its height above it. A tuning fork control (not shown) is arranged to apply a sinusoidal voltage to the tuning fork 16 and so to excite a resonant or near-resonant vibration within the *xy* plane. The probe 22 is a low-mass AFM cantilever probe and, during a scan, an interaction force is

developed between the probe tip and the sample surface. A probe detection mechanism 28 is arranged to measure the displacement of the probe tip and thus the bending of the cantilever 22, which is indicative of molecular interaction force strength. Data collected by the probe detection mechanism 28 is analysed and output to a display 30.

In general, prior art cantilever probes are fabricated from silicon or silicon nitride, which allows them to be readily produced using mature silicon microfabrication technology. Unlike prior art cantilever probes however, the probe 22 according to this invention has a polymer coating applied to the probe body. This coating, as will be explained in more detail later, serves to damp the quality factor (Q factor) of the cantilever spring.

In taking images using the apparatus 10, the sample 14 is first brought into contact with the cantilever probe 22 using the coarse driving means 20. Fine height and initial start position adjustments are made with the piezo driver 18 whilst the probe detection mechanism 28 measures the cantilever's bending as a result of the probe 22 – sample 14 interaction force. Once the measured bending reaches a desired level, the sample surface is scanned beneath the probe 22. In scanning the sample 14 under the probe 22, the tuning fork 16 is set to vibrate into and out of the plane of the Figure (*y* axis). This oscillates the stage on which the sample is mounted. At the same time, the piezo 18 translates the sample 14 in a perpendicular (*x*) direction. Sample oscillation is with a relatively large amplitude, of the order of a few microns. During the course of a scan, readings are continually taken by the probe detection mechanism 28, which, as is standard in the art, is based on an optical lever technique: cantilever bend is measured using laser light reflected from the probe. The output signal from the probe detection mechanism 28 is fed directly to a processor and display 30.

As stated above, the cantilever probe 22 shown in Figure 2a differs from

those of the prior art in that it is coated with a polymer material. The coating may be on one or both sides provided that the material itself is suitable for reducing the quality (Q) factor of the cantilever.

The Q factor is a dimensionless quantity, which may be used to quantify the dissipation (or damping) of an oscillator. It has the property that:

$$Q = \frac{\text{Energy stored in oscillator}}{\text{Energy dissipated per radian}}$$

A heavily damped system, in which stored energy is dissipated rapidly, has a low Q, and a lightly damped system has high Q. Oscillators made from Si and SiN materials do not have much internal loss and, as a result, most commercially available cantilevers will have high Q, typically of the order 50 – 500 in air. Moreover, if designed for use in tapping mode, it is advantageous for a cantilever to have a high Q. In this mode, the cantilever is driven at resonance and the interaction force measured over many cycles of oscillation. By minimising energy loss over the oscillation cycles, the high Q therefore acts as a mechanical filter.

In the case of the present invention however, it has been discovered that, contrary to the teaching of the prior art, it is desirable to use a cantilever with low Q in high-speed atomic force microscopy. If the cantilever has a high Q, it will take a long time to respond to changes and it will ring at its resonant frequency if given a stimulus, such as provided by scanning across a high feature on the sample surface. The present cantilever is designed to have a low Q by virtue of its coating. The Q factor is, ideally, sufficiently low such that any induced oscillation is critically damped. The use of a low quality factor means that little energy can be stored in the cantilever spring and so it will not "ring" for long if shocked, such as when scanning over a high region of the sample surface. This enables a speedier return to the sample surface, and consequently its better tracking

during a scan.

Many polymer materials may be used to provide the coating, and the opportunities for specific selection will be apparent to one skilled in the art. A block copolymer material in which the majority component is an amorphous rubber, with glass transition temperature just below room temperature, and the minority component is a crystalline material, coated on both sides of an AFM cantilever has been found to improve markedly its tracking capability. The copolymer was applied by solution casting. That is, a drop of solution containing the polymer is placed on the cantilever at high temperature in order to drive off the solvent. Such an arrangement has been found to permit the probe to track a sample surface even at resonant oscillation speeds such as described in WO 02/063368.

Considerations as to the polymer material and application method adopted narrow the available choice to some extent. The basic idea is to coat the cantilever with an energy-absorbing material that, ideally, does not unduly affect other cantilever properties such as mass, sharpness of tip, etc. Solution casting a cantilever with the above-described copolymer has been found to enhance energy dissipation with an acceptable increase in cantilever mass. Other coating methods can be used however. These include: "dragging" a charged polymer onto the cantilever in an electrolysis cell; chemically tagging the polymer (for instance with a thiol group) and using its reaction with the cantilever, or metal coating on the cantilever (e.g. gold in the case of thiol chemistry), to attach the polymer to the cantilever.

Applying a coating to both sides of the cantilever, given its small size, is, practically, somewhat easier to achieve than coating one side only. It is however preferred that the side of the cantilever nearer to the sample is left uncoated. The single-sided coating is sufficient to lower the Q factor and also reduces the likelihood of any coating material contaminating the sample when the probe makes contact.

Ideally the polymer material used for the coating will have a peak in its energy loss spectrum at the temperature of the probe's anticipated use and in the frequency range of the principal resonant modes of the cantilever. Typically, it should therefore be a rubbery polymer. Alternatively a copolymer or other composite with a high component of rubbery polymer may also be used.

Figure 2b shows a schematic implementation of an AFM, indicated generally by 50, that utilises a second embodiment of a cantilever probe constructed in accordance with this invention. The AFM apparatus 50 is very similar to that shown in Figure 2a, and components common to both systems are similarly referenced. As before, the plate 12 holding the sample 14 is mounted on one prong of the tuning fork 16, which is driven with a resonant or near-resonant vibration within the *xy* plane. The sample 14 (together with the plate 12 and fork 16) is scanned in three dimensions: *x*, *y* and *z* directions, with the interaction force developed being dependent both on the *xy* position of the probe 22 over the sample 14 (the pixel it is imaging), and also on its height above it. The cantilever component of the probe 22 is coated on both sides with a polymeric film and is shaped so as to have a low spring constant, less than 1 Nm^{-1}. Unlike the cantilever shown in Figure 2a however, the probe 22 according to this embodiment of the invention has a magnetic bead 24 mounted above the tip. A magnet 26 is incorporated within the AFM to provide a magnetic field of sufficient strength to exert a force on the magnetic bead 24. The probe detection mechanism 28 is arranged to measure the bending of the cantilever 22, as for the apparatus 10 shown in Figure 2a. Data collected by the probe detection mechanism 28 is analysed and output to a display 30.

In taking images using the apparatus 50, the contact mechanism to establish an interaction force and scanning technique are substantially as described in relation to the apparatus 10 of Figure 2a. Once the desired level of interaction force, and hence cantilever bend, is established

however, then the magnet 26, which is not present in the Figure 2a apparatus 10, is switched on and a magnetic field *B* is generated in the vicinity of the probe tip. The magnetic bead 24 interacts with this field, which is directed such that the resultant magnetic force attracts the magnetic bead 24 downwards into the sample 14. The probe tip is therefore held in contact with the sample 14 by the direct action of this magnetic force. With the magnetic field on, the sample surface is oscillated (at resonance) and scanned beneath the probe 22 and the output signal processed as before.

In order to appreciate the features that are necessary to this invention it is helpful to look at a diagrammatic representation of the forces involved while a scan is being performed. This is illustrated in Figure 3, which shows the same set up as Figure 1 and so like components are similarly referenced. With reference to Figure 3, there is shown a sample 1 that is being scanned by a probe of an atomic force microscope (AFM) in accordance with the present invention. The probe comprises a substrate 2 from which a cantilever 3 extends, the cantilever 3 having a sharp probing tip 4 mounted at an end remote from the substrate 2. In preparation for a scan, a downwards force ($F_{external}$) is applied to the probe at its substrate end 2 via its mounting to the AFM, moving the probe tip 4 into contact with the sample 1. In order to maintain contact for the duration of a scan, the force $F_{external}$ is greater than that required simply to bring the tip 4 into contact with the sample 1. As a result the cantilever 3 is bent upwards from its rest position 5 as the sample is scanned. As before, a force proportional to *kx* is generated as a result of the cantilever bending and directs the probe tip 4 downwards towards the sample surface.

In the event that a probe designed in accordance with the present invention is deflected from the sample surface, for example by encounter with a raised portion, two factors assist in restoring it back towards contact. This enables better tracking of the surface to be achieved, even at high scan

speeds. First, the Q factor of the cantilever is low. Stored energy is rapidly dissipated and the bending force, originally proportional to kx, is reduced quickly as the probe straightens, with minimal ringing. Secondly, as is seen more clearly in the embodiment shown in Figure 2b, a second attractive force F_{direct} between probe and sample also assists in straightening the probe and so reducing the bending force kx.

The total restoring force holding the probe to the surface is now:

$$F_{direct} + \alpha kx,$$

where α is a constant of proportionality for the downwards force arising from the cantilever bend. Ideally, the additional force F_{direct} is greater than the cantilever bending force kx. Its magnitude should moreover be sufficiently large to bring the probe into contact with the surface, should it lose contact, within approximately one pixel.

In the embodiment depicted in Figure 2b, the additional force F_{direct} is a magnetic force, provided by applying a magnetic field to a cantilever tip that incorporates a magnetic bead. Clearly therefore the positioning of the magnet within the AFM is not critical, it merely has to be arranged such that there is a downward force component pulling the probe tip 4 into the sample 1.

In the embodiment depicted in Figure 2a, the additional force F_{direct} is still contributing to the tracking performance of the cantilever, but its origin is more subtle. As the probe and sample are brought into close proximity a capillary neck is generally believed to form, connecting the two. This capillary neck is thought to arise from fluid that will inevitably be present in the sample environment when it is imaged in air, which condenses about the probe – sample contact. In normal operation, it is found that the direct force F_{direct} arising from the capillary neck is sufficiently large that it quickly

forms the dominant restoring force on the low-Q cantilever i.e. $F_{direct} > kx$. This is particularly true for hydrophilic surfaces.

Regardless of the origin of the additional direct force F_{direct}, the low Q of the cantilever permits stored energy to be dissipated rapidly as the cantilever is straightened and restored to its contact with the surface by the action of the direct force F_{direct}. Tracking of the sample surface by the cantilever is therefore achieved by a kind of mechanical feedback loop, which is faster acting than the prior art tracking mechanisms with their dependency on the cantilever bending force kx.

In order to assist in achieving $F_{direct} > kx$, the cantilever should be further designed with a relatively low spring constant. Typically this should be less than 1 Nm^{-1}, which can be achieved by using a suitably shaped cantilever. In the present invention, the cantilever force kx is useful only to define the position in space at which the probe sits, i.e. the interaction force between probe and sample, and so to enable an image to be collected.

The ability to exploit a direct restoring force F_{direct} as opposed to relying on the cantilever force in sample tracking represents a significant improvement over the prior art. By providing a cantilever with low Q, the cantilever contribution kx to the restoring force is dissipated rapidly, and the predominant restoring mechanism is the direct force F_{direct}. This applies regardless of whether the direct force is a "natural" force, generated by means of the capillary neck, or an additional, external force, such as that applied via a magnetic bead. In either case, the restoring force has a magnitude that is essentially independent of the position of the cantilever. By way of contrast, the magnitude of the prior art restoring force kx depends on the displacement x of the cantilever from its rest position. Thus high restoring forces are generated at particularly high regions of the sample. This may restore the probe to its surface position quickly, but such rapid movement may also damage the sample. It is very difficult to ensure

consistently that samples are not damaged if the restoring force is permitted to vary in this manner. A restoring mechanism implemented in accordance with this invention has a magnitude that is effectively independent of sample height.

Consider now the embodiment of the invention shown in Figure 2b. It is not essential that the applied force is a magnetic force, although it is preferred that it is a force whose magnitude does not depend on sample height. An alternative to the magnetic bead / magnet arrangement shown is, for example, to incorporate an electrostatically charged region in the probe that is attracted towards the sample by an electric field. It is further required that the applied force F_{direct} is larger than the cantilever restoring force kx, at least within a certain time period after the start of energy dissipation (i.e. when contact is first lost with the surface). That is to say, it is required that sufficient energy is lost from the cantilever system within the time taken for the scanning apparatus to move, say, a few image pixels, such that the direct force F_{direct} is the dominant restoring force. The larger the direct force F_{direct} therefore, the less strict is expected to be the requirement for low Q.

The cantilever of the present invention is selected to have a low Q, ideally such that any induced oscillation is critically damped. As described herein, the most preferred arrangement, and one which is sufficiently effective to enable improved tracking by means of the natural restoring force due to the capillary neck, is to coat one or both sides of the cantilever with an energy absorbing material, such as a polymer film. An alternative, particularly if a large magnetic (or other additional) force is applied, means to ensure low Q is by judicious selection of cantilever shape. Another alternative is simply to provide a low Q factor by immersing the cantilever in liquid during the scan. In this situation Q ~ 1.

The cantilever, probe tip and any additional component such as the

magnetic bead are ideally of low mass. This naturally increases the acceleration of the tip back to the surface for a given restoring force and so better enables the probe to track the surface.

It is to be noted that the apparatus shown in Figures 2a and 2b is merely illustrative of an exemplary AFM. There are numerous different embodiments of AFM with which this invention may be implemented. For example, the piezos 18, 20 may be arranged to translate the probe 22 rather than the sample 14, all that is required is that a relative translation is imparted by the scan. Nor is mounting on a tuning fork necessary. This arrangement is simply used in this embodiment in order to illustrate the applicability of this invention to fast scanning techniques that make use of a resonant oscillation. It is equally applicable to slower scanning methods. The probe 22 may alternatively be oscillated in place of the sample 14, although this may cause problems when measuring probe deflection using the optical lever technique.

Cantilever deflection may be measured by means other than the optical lever technique. Alternative techniques known in the art include interferometry and piezoelectrically coated cantilevers.

If a tuning fork 16 is used then it may be one of a number of commercially available forks, or of bespoke design to provide a desired frequency of oscillation. A suitable example is a quartz crystal fork with resonant frequency of 32 kHz. A tuning fork is well suited to this application as it is designed with highly anisotropic mechanical properties. Its resonances are therefore independent and can be individually excited and so limited to only that (or those) in the plane of the sample. Importantly, the fork 16 can be resonated in one direction and scanned in another, without coupling occurring between modes. It therefore permits stable fast motion of the sample 14 as it is interrogated by the probe 22. Alternative mechanical resonators that have a similar facility for well-separated lateral and vertical

resonances can be used in place of the tuning fork.

The invention is not limited to pure AFM operation, although it is required that there is a force interaction between the probe and the sample surface. This mode of operation can however be combined with microscope components designed to monitor other interactions or interaction indicators between probe and sample. Examples of other interactions may include optical, capacitative, magnetic, shear force or thermal interactions. Other indicators include oscillation amplitude, either tapping or shear force, capacitance or induced electric currents. These various modes of operation of general probe microscopes are described, for example, in UK patent application number 0310344.7.

The interaction of the probe with the sample surface that is exploited in AFM also makes it possible to affect the properties of the surface and so deliberately to "write" information to the sample. This technique is known as nanolithography, and AFMs are widely used for this purpose. For example, by application of a voltage to a conductive cantilever a region of a metallic layer of a sample wafer can be oxidised. Another example exploiting two-photon absorption and polymerisation of a photoresist is described in "Near-field two-photon nanolithography using an apertureless optical probe" by Xiaobo Yin *et al.* in Appl. Phys. Lett. 81(19) 3663 (2002). In both examples the very small size of the probe enables information to be written to an extremely high density. The AFM and cantilever probe of this invention can also be adapted for use in nanolithography. The ability to improve surface tracking with this invention not only offers the potential for faster writing times than previously achieved, but also offers the potential for increased image resolution i.e. write density. To render it more adapted for use in nanolithography the probe tip may be electrically conductive, it may be metal coated in order to increase its optical interaction with the surface or it may be coated with selected molecular species for use in dip pen lithography applications.

<u>CLAIMS</u>

1. A probe (22) for use in an atomic force microscope or for nanolithography, the probe comprising a cantilever (3) and sharpened tip (4) characterised in that the cantilever (3) is coated on at least one side by an energy-absorbing material.

2. A probe (22) according to claim 1 characterised in that the energy-absorbing material is a polymer film.

3. A probe (22) according to claim 2 characterised in that the polymer film is formed of a copolymer with majority component that is an amorphous rubber and a minority crystalline component.

4. A probe (22) according to claims 2 or 3 characterised in that the cantilever (3) is coated with polymer by solution casting.

5. A probe (22) according to any preceding claim characterised in that the cantilever (3) is coated on both sides.

6. A probe (22) according to any preceding claim characterised in that it additionally includes a magnetic element (24) located on the cantilever (3) in the region of the tip (4).

7. A probe (22) for use in an atomic force microscope or for nanolithography, the probe comprising a cantilever (3) and sharpened tip (4) characterised in that the probe also includes a magnetic element (24) located on the cantilever (3) in the region of the tip (4).

8. A probe according to claim 7 characterised in that the cantilever (3) has a low quality factor.

9. A probe according to claim 8 characterised in that the cantilever (3) is coated on at least one side with a polymeric film.

10. An atomic force microscope (10) for imaging a sample (14) in accordance with a molecular interaction force between the sample (14) and a cantilever probe (22), the microscope (10) comprising

driving means (16, 18, 20) arranged to provide relative scanning motion between the probe (22) and the sample surface and capable of bringing the sample (14) and probe (22) into close proximity, sufficient for a detectable interaction to be established between them; and

a probe detection mechanism (28) arranged to measure deflection of the cantilever probe (22);

characterised in that, the microscope (10) includes the probe (22) of any one of claims 1 to 6.

11. An atomic force microscope (10) for imaging a sample (14) in accordance with a molecular interaction force between the sample (14) and a cantilever probe (22), the microscope (10) comprising

driving means (16, 18, 20) arranged to provide relative scanning motion between the probe (22) and the sample surface and capable of bringing the sample (14) and probe (22) into close proximity, sufficient for a detectable interaction to be established between them; and

a probe detection mechanism (28) arranged to measure deflection of the cantilever probe (22);

characterised in that, the microscope (10) includes direct force (F_{direct}) generating means (24, 26) arranged such that, in operation, a direct

force (F_{direct}) is applied to the sample (14), the probe (22) or between the two, the force (F_{direct}) being directed so as to attract the probe (22) towards the sample (14) or *vice versa*.

12. A microscope according to claim 11 characterised in that the direct force (F_{direct}) has magnitude that is independent of the degree of deflection of the cantilever probe (22).

13. A microscope according to claim 12 characterised in that the cantilever probe (22) has spring constant k and the probe (22) properties and direct force (F_{direct}) are arranged such that, at least within a predetermined timescale, the additional force (F_{direct}) is greater than the restoring force kx provided by a cantilever deflection x as it scans the surface of the sample (14).

14. A microscope according to claim 13 characterised in that the probe (22) has spring constant k that is less than 1 Nm^{-1}.

15. A microscope according to any one of claims 11 to 14 characterised in that the probe (22) comprises a cantilever (3) with low quality factor.

16. A microscope according to claim 15 characterised in that the probe (22) and sample (14) are immersed in liquid during operation of the microscope.

17. A microscope according to claim 15 characterised in that the cantilever (3) is coated on at least one side with a polymeric material.

18. A microscope according to claim 17 characterised in that the direct force (F_{direct}) generating means (24, 26) comprises a capillary neck formed between probe (22) and sample (14).

19. A microscope according to any one of claims 11 to 17 characterised in

that the direct force (F_{direct}) generating means (24, 26) comprises a magnet (26) and a magnetic element (24) incorporated in the probe (22).

20. A method of collecting image data from a scan area of a sample (14) with nanometric features wherein the method comprises the steps of:-

    (a) Moving a cantilever probe (22) coated on at least one side with an energy-absorbing material and with a tip of sub-nanometric dimensions into close proximity with a sample (14) in order to allow an interaction force to be established between probe (22) and sample (14);

    (b) Permitting a direct force (F_{direct}) to be established between sample (14) and probe (22) such that the probe (22) is encouraged to move towards the sample (14) or *vice versa*;

    (c) Scanning either the probe (22) across the surface of the sample (14) or the sample (14) beneath the probe (22) whilst providing a relative motion between the probe (22) and surface such that an arrangement of scan lines covers the scan area;

    (d) Measuring deflection of the cantilever probe (22); and

    (e) Processing measurements taken at step (d) in order to extract information relating to the nanometric structure of the sample.

21. A method of collecting image data from a scan area of a sample (14) with nanometric features wherein the method comprises the steps of:-

    (a) Moving a cantilever probe (22) with tip of sub-nanometric dimensions into close proximity with a sample (14) in order to allow an interaction force to be established between probe (22) and

sample (14);

(b) Applying a direct non-contact force (F_{direct}) to either the sample (14) or the probe (22) such that the probe (22) is encouraged to move towards the sample (14) or *vice versa*;

(c) Scanning either the probe (22) across the surface of the sample (14) or the sample (14) beneath the probe (22) whilst providing a relative motion between the probe (22) and surface such that an arrangement of scan lines covers the scan area;

(d) Measuring deflection of the cantilever probe (22); and

(e) Processing measurements taken at step (d) in order to extract information relating to the nanometric structure of the sample.

22. A scanning probe microscope (10) for writing information to a sample (14) by means of an interaction between the sample (14) and an AFM cantilever probe (22), the microscope comprising

driving means (16, 18, 20) arranged to provide relative scanning motion between the probe (22) and the sample surface and capable of bringing the sample (14) and probe (22) into close proximity; and

a probe writing mechanism arranged to vary intermittently, on a timescale shorter than one scan line, the strength of the interaction between the probe and the sample and so to change intermittently a property of the sample surface in the locality of the probe;

characterised in that, the microscope includes the probe (22) of any one of claims 1 to 6.

23. A scanning probe microscope (10) for writing information to a sample

(14) by means of an interaction between the sample (14) and an AFM cantilever probe (22), the microscope comprising

driving means (16, 18, 20) arranged to provide relative scanning motion between the probe (22) and the sample surface and capable of bringing the sample (14) and probe (22) into close proximity; and

a probe writing mechanism arranged to vary intermittently, on a timescale shorter than one scan line, the strength of the interaction between the probe and the sample and so to change intermittently a property of the sample surface in the locality of the probe;

characterised in that, the microscope includes direct force (F_{direct}) generating means (24, 26) arranged such that, in operation, a direct force (F_{direct}) is applied to either the sample (14) or the probe (22), the force (F_{direct}) being directed so as to attract the probe (22) towards the sample (14) or *vice versa*.

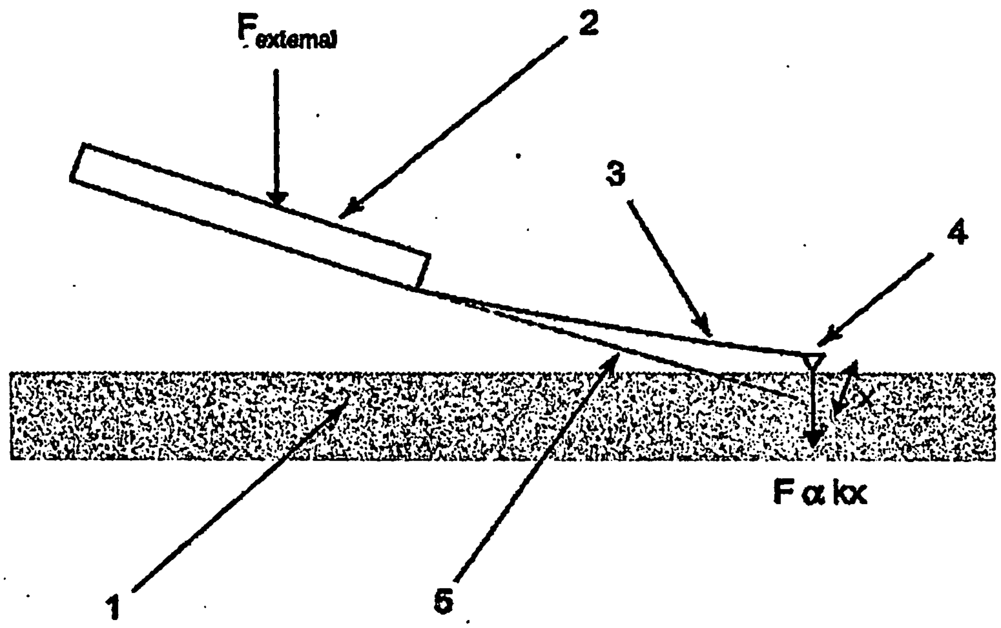

Fig 1

Prior Art

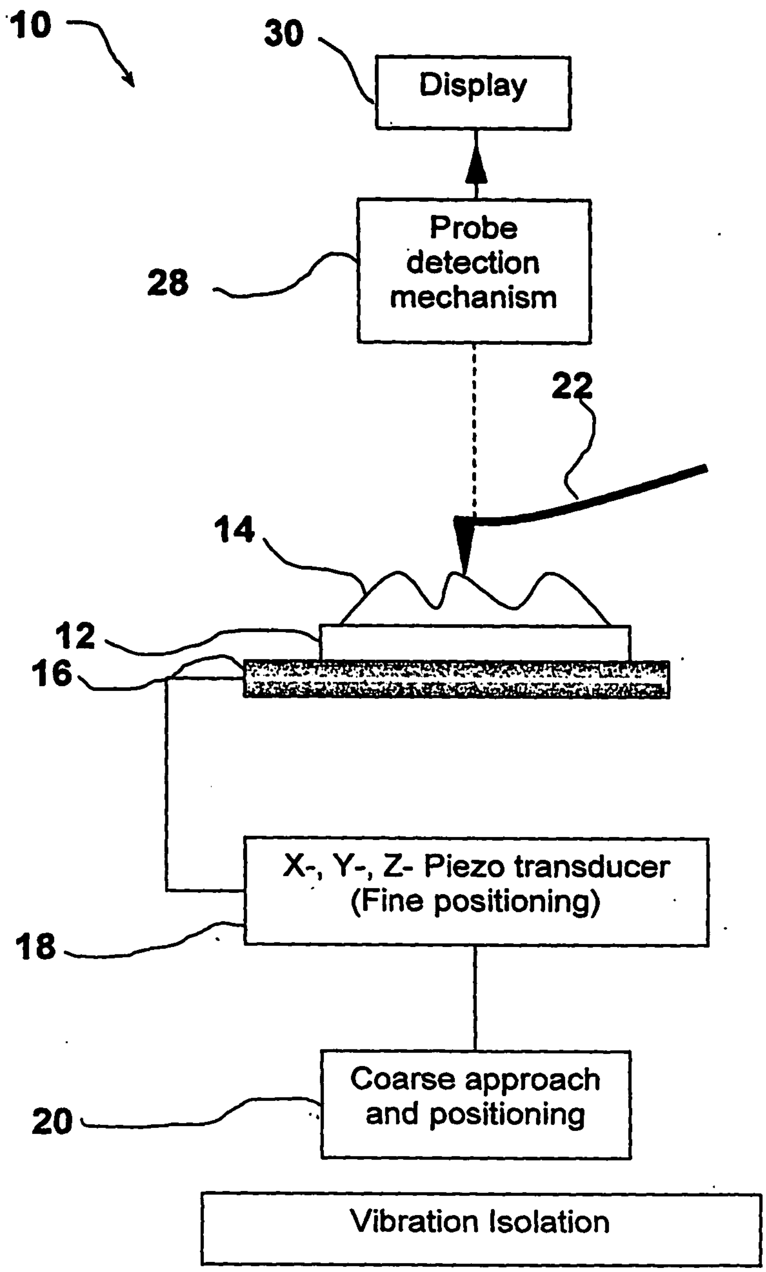
10
30
Display
Probe detection mechanism
28
22
14
12
16
X-, Y-, Z- Piezo transducer (Fine positioning)
18
Coarse approach and positioning
20
Vibration Isolation

Fig 2a

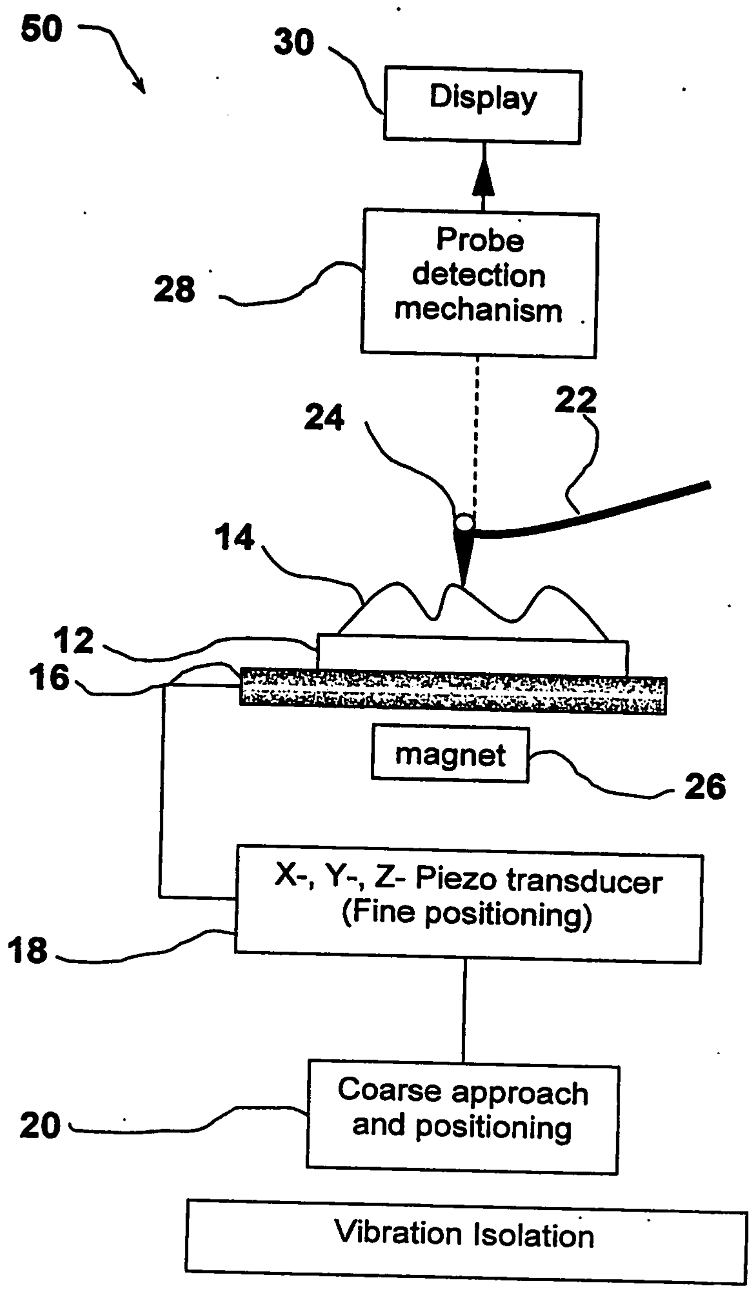
50
30
Display
Probe
detection
mechanism
28
24
22
14
12
16
magnet
26
X-, Y-, Z- Piezo transducer
(Fine positioning)
18
Coarse approach
and positioning
20
Vibration Isolation

Fig 2b

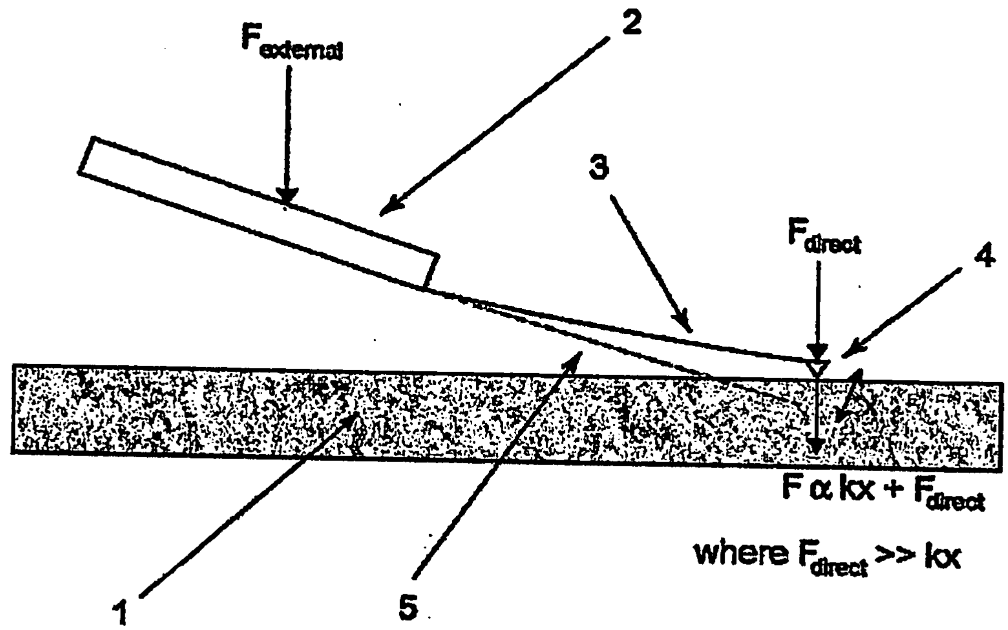
$F_{external}$
2
3
F_{direct}
4
$F \alpha\ kx + F_{direct}$
where $F_{direct} >> kx$
1
5

Fig 3